

Process Equipment Design
Prof. Shabina Khanam
Department of Chemical Engineering
Indian Institute of Technology – Roorkee

Lecture –42
Design of Crystallizer: Types

Hello everyone. Welcome to the second lecture of 9th week of the course Process Equipment Design and in this lecture I will discuss different types of crystallizer. In previous lectures, we have defined the crystallization, we have discussed how to design a crystallizer and then design of crystallizer is illustrated with the help of examples and now we will focus on different types of crystallizer which are used in the industry. So, let us focus on the crystallization equipment first.

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Crystallization Equipment

Satisfactory operation of a crystallizer depends upon quite a few factors:

- The rates of nucleation and crystal growth both depend upon the extent of super saturation of the solution. At a high super saturation level, the rate of nucleation is high. So there is a possibility of a higher fraction of fines in the product.
- At a low level of super saturation, the rate of nucleation is slow. So it is likely that the product will contain a higher fraction of larger size crystals.
- The rate of secondary nucleation can be reduced (thereby reducing the fraction of fines in the product) by maintaining a low mechanical energy input.

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So, as far as satisfactory operation of crystallizer is concerned there are different factors on which it depends and these factors are the rate of nucleation and crystal growth both depend upon the extent of super saturation of the solution. So, how much super saturation is there the crystal formation depends on that. At a high super saturation level the rate of nucleation is high.

So, there is a possibility for a higher fraction of fine in product. So, if nucleation will be higher in that case we can have small size particles or small size crystals and their fraction is more when super saturation level is high. So, if you consider the super saturation level is high

it means that formation of crystal will be very fast. So, in that case fine size crystals amount will be more.

However, on the other side if low level of super saturation is considered in that case nucleation rate will be less. In that case, the residence time for the solution will be more and solution gets more and more time to form crystal. In that case usually crystal size are larger. So, in that case we have high friction of large crystals and further the rate of secondary nucleation can be reduced.

Thereby, reducing the fraction of fines in the product by maintaining low mechanical energy input. So, when we are not agitating it much it means we can obtain large size crystal because agitation will reduce the crystal size.

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Crystallization Equipment

Mixed Suspension Mixed Product Removal (MSMPR) Crystallizers or Circulating Magma Crystallizers

These are the most important class of industrial crystallizers. The growing crystals are kept in suspension by agitation; 20 – 40% solids in suspension is common. Two types of crystallizers of this class are described here:

- Forced-circulation (evaporative) crystallizer ✓
- Draft-tube-baffle (DTB) crystallizer ✓

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And let us see the equipment which are used in industry for crystallization. So, here initially I am considering mixed suspension, mixed product removal crystallizer for circulating magma crystallizer. So, these are most important class of industrial crystallizer. The growing crystal are kept in suspension with agitation and 20% to 40% solids in the suspension is common. So, in this category we usually have two types of crystallizer.



The first one is forced circulation and that is evaporative crystallizer and second is draft tube baffle crystallizer. So, let us focus on the working of these one by one.

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Crystallization Equipment

Forced-circulation (evaporative) crystallizer

A forced circulation (FC) crystallizer has four major components. The slurry is pumped through an external steam-heated vertical heat exchanger to raise its temperature by 2 to 6°C. Heat exchanger tube size of 1.25 to 1.75 inch is common; the liquid velocity is maintained at 2-3 m/s. No boiling occurs in the heat exchanger. The hot liquid is thrown into the crystallizer body when evaporation of the liquid occurs. This raises the solution concentration, reduces its temperature (because of evaporation of some liquid) and generates the supersaturation required for crystal growth.



<https://www.chemicalprocessing.com/articles/2003/381/>

So, first of all I am focusing on forced circulation evaporative crystallizer. And as the name says it is forced circulation means it is using a pump to inlet the feed. So, feed is basically entering from here and it is mixed with the liquor which is exiting the crystallizer and both will enter into the heat exchanger. Here, temperature of the feed is increased, but not evaporation here only the sensible heat transfer occurs.

And from this recirculation pipe it is entering to the crystallizer body tangentially and here evaporation takes place. Such type of working we have also discussed previously. So, whatever vapour is generated it is entering into the condenser and here we have the formation of the crystal which comes down using swirl movement and further we can take the product out from this discharge.

So, a forced circulation crystallizer has four major component. The slurry is pumped through an external steam heated vertical heat exchanger to raise its temperature from 2 to 6 degree Celsius. Heat exchanger tube size is usually 1.25 to 1.75 inch, liquid velocity is usually maintained as 2 to 3 meter per second, no boiling occurs in the exchanger that is why I have told that only sensible heat transfer will occur.

Hot liquid is thrown into the crystallizer body when evaporation of the liquid occurs and raise the solution concentration and so the crystal formation occur when the solution reaches to super saturation condition.

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Crystallization Equipment

Forced-circulation (evaporative) crystallizer

A condenser at the top removes the vapour generated. The equipment may be put under vacuum by coupling a barometric leg to the condenser. The product is withdrawn from the circulating pipe. The feed also enters the crystallizer through this pipe, but at a lower level as shown in Figure.

The pump speed should be low in order to reduce the mechanical energy input so that the secondary nucleation rate by contact or attrition remains small.

Many inorganic salts such as ammonium sulphate, sodium chloride, trisodium phosphate, potassium nitrate, citric acid, sugar, etc. are crystallized in an FC crystallizer. Crystals typically of the size range 30-60 mesh are produced.

So, in this way forced circulation crystallizer works and here we have some more points about this that is a condenser at top removes the vapour generated. The equipment maybe put under vacuum by coupling a barometric leg of the condenser. The product is withdrawn from the circulating pipe that we have discussed already and feed also enters the crystallizer through pipe, but at lower level as you can see in this diagram where discharge is here, but feed is entering at lower level through the circulating pipe only.

So, further we have to consider that pump speed should be low in order to reduce the mechanical energy input and so the secondary nucleation rate by contact or attrition remains small. So, in this way we consider the working of forced circulation crystallizer. Now we have some examples where it is used. It is most commonly used in ammonium sulfate crystal formation, sodium chloride, trisodium phosphate, potassium nitrate, citric acid, sugar etcetera.



And in this case usually the crystal size are 3 to 60 mesh. So, that is basically forced circulation crystallizer.

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Crystallization Equipment

Draft-tube-baffle (DTB) crystallizer

The body of a DTB crystallizer is provided with an inner baffle tube and also a skirt baffle. A long-shaft slow-moving impeller throws the liquid upwards through the baffle tube towards the surface. This causes circulation of the magma in the crystallizer body; more circulation than in an FC unit is achieved at the same power input. Fouling is also less than that in an FC. The magma also flows out of the body through an annular zone between the skirt baffle and the wall, enters a steam-heated exchanger and recycles back to the crystallizer vessel.

Now, we have draft tube baffle crystallizer. In this case this is the schematic you see here we have this structure that is nothing, but the tube and this we call as the draft tube and as far as baffle are concerned this is basically the baffle. So, in this case what will happen heated feed enters the center of the crystallizer and where we are having the draft tube. So, it is basically entering into the draft tube through this impeller the movement of liquid is in this direction.

So, as it is moving in this direction vapour formation occur and as it is moving in this direction vapour formation occurs and at the same time crystals are also formed and that crystal basically travels in this solution. Vapour moves up and whatever crystals are there because of this impeller it is moving in this direction. And further we have the space between draft tube and baffle and from here crystals are basically moves downwards.

However, in some cases crystals are also moving with the liquid in this direction and in this direction when the size of the crystals are very small. The larger size crystals may settle from this direction, however, smaller size crystals are basically entering and revolving with this solution. So, when the mother liquor which is leaving the system the small size crystals are already available with this mother liquor.

And that is mixed with the feed and recycles back to the heat exchanger and so the crystallizer and the product is discharged from this elutriating leg and so we can have the crystal formation. However, in this case crystals are also available with mother liquor not in the form of solute, but in the form of small crystals only. So, that recycles back to the heat

exchanger and that is mainly occurring because we have longer path and that longer path is because of this draft tube baffle as well as the baffle which we consider as the skirt baffle.

So, this is basically the skirt baffle. So, in this way it is working. So, let us discuss some points about this a few of them I have already discussed so let us start that. So, the body of DTP crystallizer that is the draft tube baffle crystallizer, it provides the inner baffle tube as well as the skirt baffle I have already told you. Long shaft slow moving impeller which is available at the center it throws the liquid towards the surface.

And this causes circulation of magma in the crystallizer body and in this case we can obtain more circulation in this crystallizer in comparison to forced circulation unit and because we are having more circulation over here this has less tendency to create fouling in the equipment. Magma also flows out of the body through annular zone between skirt baffle and the wall that is basically this section and it is further entering into the steam heated exchanger.

So, usually what happens mother liquor contains solvent along with the solute, but that solute is mixed form in the solvent, but here we can have the clear liquid along with the fine crystal it means that is basically the magma. So, some part of the magma which is exiting the crystallizer it is mixed with the feed and then recycles back to the crystallizer.

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Crystallization Equipment

Draft-tube-baffle (DTB) crystallizer

Some of the fines may dissolve in the heat exchanger. Thus the crystallizer has a fine destruction feature. Settling of large crystals occurs in the annular region so that only the fines leave with the recirculating slurry. However, this happens only when the mother liquor and the crystals have sufficiently different densities. An elutriating leg can be fitted to the conical bottom to achieve further classification of the product.

A DTB crystallizer controls the solution superheating at the boiling surface to within 2°F and thereby controls the bulk liquid supersaturation within a range which is attained in an FC crystallizer. A close control of crystal size is thus possible. Crystals ranging from 8-30 mesh may be produced by controlling the process conditions. Typical applications are for crystallization of adipic acid, KCl, K₂SO₄, etc.

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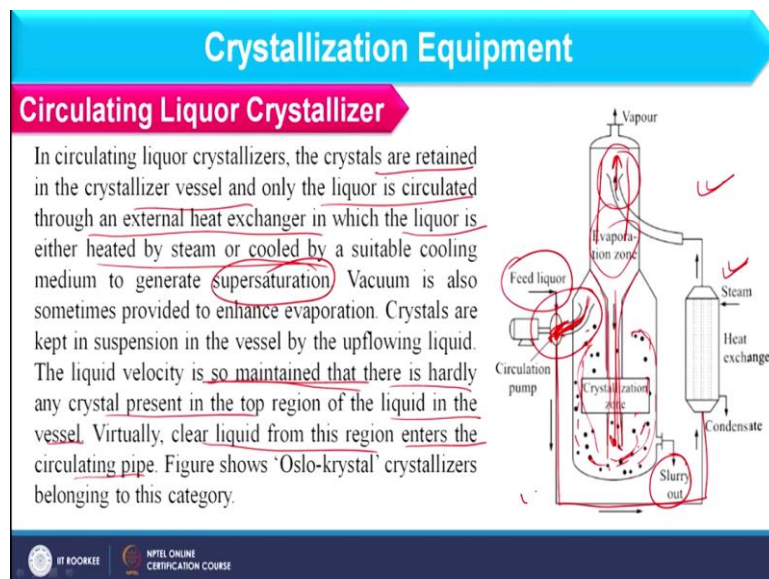
So, here I am having some more points about this. Some of the fines may dissolve in the heat exchanger and thus the crystallizer has a fine destruction feature because of the fine crystals.

Settling of large crystals occur in the annular region so that the fines leave with the recirculating slurry. However, this happens only with the mother liquor and the crystal have sufficiently different density.

And elutriating leg can be fitted into the conical bottom to achieve further classification of the product that point we have already shown in the schematic. And in this type of crystallizer it controls the solution superheating at the boiling surface to within 2 degree Fahrenheit and thereby controls the bulk liquid super saturation within a range which is attain in forced circulation crystallizer. A close control of crystal size is thus possible.

Usually the range is from 8 to 30 however in forced circulation it is from 30 to 60 mesh. So, as far as applications are concerned these type of crystallizers are used to produce potassium chloride and potassium sulfate crystals. We have few more crystallizer and in that category we have circulating liquor crystallizer.

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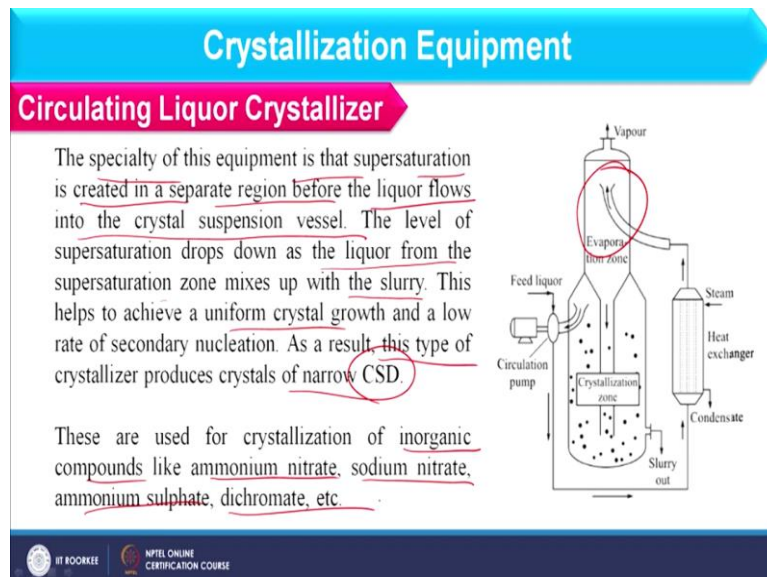
The schematic is shown over here. So, if you see here I am having the feed which is mixed with the liquor which is coming out from the crystallizer that is the mother liquor and it is mixed over here and then it is entering into the heat exchanger. So, superheated solution is entering into the vapour zone of this crystallizer and here evaporation takes place. So, in this case once evaporation happens because of the vacuum in the system the crystals and liquid is moving down.

And crystal are usually collected at the bottom section because of this long tube liquid is coming with high velocity in this region and vapour which is exiting the system contains almost negligible amount of liquid droplets. So, in that case crystals are basically settles down and from here can have the slurry out. So, in that case the liquor is basically circulating not the magma as we have discussed in DTB crystallizer.

So, in this type of crystallizer crystals are retained in crystallizer vessel and only liquor is circulated through the external heat exchanger in which liquor is either heated by the steam or cooled by suitable cooling media to find out super saturation condition. The liquid velocity is so maintained that there is hardly any crystal present in the top region of the liquid in the vessel.

So, you can see with the vapour no crystal as well as no liquid droplet is there because it has sufficient height for disengagement. So, virtually clear liquid from this region enters the circulating pipe that is basically this region and this crystallizer is basically Oslo krystal crystallizer.

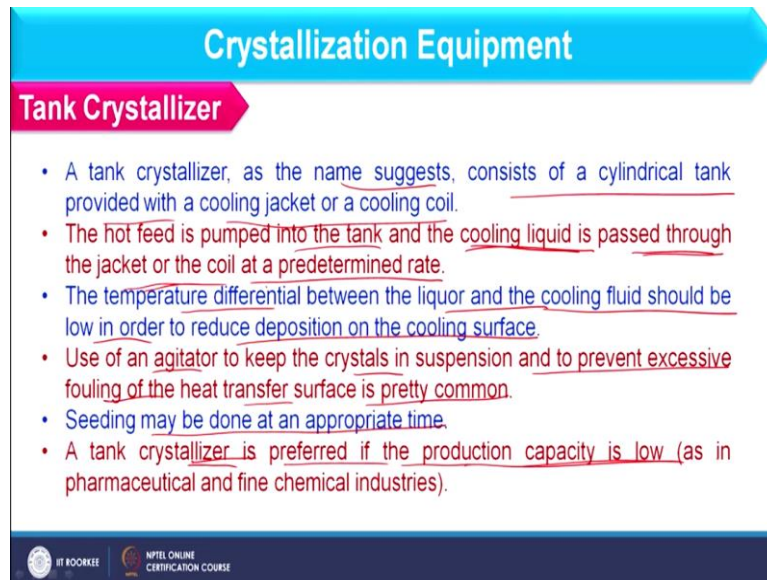
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So, in this case the specialty of this equipment is that super saturation is created in a separate region before the liquor flows into the crystal suspension vessel. So, you can see here we can have the super saturation condition the level of super saturation drops down as the liquor from the super saturation zone mixes up with the slurry. So, this helps to achieve a uniform crystal growth and a low rate of secondary nucleation.

So, as a result this type of crystallizer produces crystals of narrow crystal size distribution and these are specifically used to make the inorganic compound such as ammonium nitrate, sodium nitrate, ammonium sulfate and dichromate. So, in this way circulating liquor crystallizer works and now we will focus on tank crystallizer.

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Crystallization Equipment

Tank Crystallizer

- A tank crystallizer, as the name suggests, consists of a cylindrical tank provided with a cooling jacket or a cooling coil.
- The hot feed is pumped into the tank and the cooling liquid is passed through the jacket or the coil at a predetermined rate.
- The temperature differential between the liquor and the cooling fluid should be low in order to reduce deposition on the cooling surface.
- Use of an agitator to keep the crystals in suspension and to prevent excessive fouling of the heat transfer surface is pretty common.
- Seeding may be done at an appropriate time.
- A tank crystallizer is preferred if the production capacity is low (as in pharmaceutical and fine chemical industries).

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So, as far as this tank crystallizer is concerned as the name suggests it consists the cylindrical tank provided with the cooling jacket or cooling coil. So, usually it is a simple tank where either we can provide jacket or the cooling coil inside this. So, in this way it is called a tank crystallizer. So, the hot feed is pumped into the tank and cooling liquid is passed through the jacket or the coil at a predetermined rate.

As we have discussed already that how the jacket will be placed and how the cooling coil will be placed. So, in this case the super saturation condition is usually obtain by cooling not evaporation as we have discussed in earlier crystallizer. So, in this case temperature differential between liquor and the cooling fluid should be low in order to reduce deposition on the cooling surface so that we should avoid.

Further, use of an agitator to keep the crystals in suspension and to prevent excessive fouling of the heat exchanger surface is pretty common. So, because in this crystallizers recirculation is not occurring it means fluid is not moving. Fluid is usually at stagnant condition. So, to suspend the crystal in the solution it is better to use agitator otherwise crystal will be deposited here and there and fouling will also be there.

So, agitation is must in this type of crystallizers and seeding maybe done at an appropriate time because if you consider this tank crystallizer it is basically batch type of crystallizer. So, in that case we should provide some seeding and after that the crystal will be formed over those seeds. Further, tank crystallizer is preferred if production capacity is low as we consider in pharmaceutical and chemical industry.

But for low capacity this type of crystallizers can be used. So, you consider this is the simplest type of crystallizer, but it will be used for lesser capacity of course and it will include lesser cost also.

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Crystallization Equipment

Other Types of Crystallizers

Besides the above types of crystallizers, many other designs of the equipment are in use. For viscous and fouling solutions, 'scraped-surface crystallizers', having a coaxial double-pipe construction and provided with a screw-type internal surface scraper, are convenient. Supersaturation is generated by cooling. These crystallizers are usually of small capacity because of the limited heat transfer area. The crystal size distribution is generally wide.

Common examples of scraped-surface crystallizers are the Votator and Armstrong crystallizer and the Swenson Walker crystallizer.

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And we have some other type of crystallizer as besides the above type of crystallizer many other designs of the equipment are in use for viscous and fouling solution, scraped surface crystallizers are used which is having coaxial double pipe construction and provided with the screw type internal surface scrap. So, usually it is basically double pipe (()) (20:09) and at this outer surface of inner pipe we have this type of blade.

So, because of this the scraping section occurs in annular section of the double pipe unit. So, in this case super saturation is generated by cooling. So, in this case evaporation does not happen we consider cooling system. These crystallizers are usually of small capacity because of the limited heat transfer area. Crystal size distribution is generally wide. Commonly example of scrapped surface crystallizers are votator and Armstrong crystallizer and the Swenson walker crystallizer. So, these are some of the examples.

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Crystallization Equipment

Other Types of Crystallizers

These crystallizers are mainly used for crystallizing organic compounds like fatty acids, dyes, p-xylene, chlorobenzene, naphthalene, etc. and also for some inorganic materials that have a tendency to form scale on surfaces.

Fluidized bed crystallizers, surface-cooled crystallizers, direct-contact refrigeration crystallizers are a few other types in use.

Application of ultrasound has been found to be effective for controlled nucleation and has the potential of improving CSD and product quality of industrial crystallizers.

And further we consider that these crystallizers are mainly used for crystallizing organic compound such as fatty acid, dyes, p-xylene, chlorobenzene, naphthalene etcetera and also for some inorganic material that have tendency to form scale on the surface. And that scale on the surface can be removed by scrapping action. Fluidized bed crystallizers, surface cooled crystallizer, direct contact refrigeration crystallizers are other type of crystallizer which are in used in industry.

So, apart from that application of ultrasound has been found to be effective for controlled nucleation and has the potential of improving crystal size distribution and product quality of industrial crystallizer because that ultrasound is basically creates the cracks in the crystal. So, crystal size distribution can be improved and with this impurities which are associated with the crystal can be removed through ultrasound action and so product quality can also be improved.

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Crystallization Equipment

A Few Common Operational Problems

- Industrial crystallization is a complex process with associated operational problems. The more common problems are low yield, more fines and a wide size distribution, and fouling of the vessel and the heat transfer surface.
- Some of these problems can be solved by properly controlling the supersaturation and magma density and by adjusting the average residence time of the crystals.
- Besides deteriorating the product quality, a larger fraction of fines is likely to cause deposition on the surfaces leading to 'incrustation'.
- If the yield is poor, a part of the mother liquor is recycled after separation of the crystals.
- A high crystal growth rate may adversely affect the purity since some of the foreign substances may get entrapped within the crystals.



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So, as far as crystallization equipment is concerned we have few common operational problems such as industrial crystallization is a complex process with associated operational problems. And the more common problems are low yields more fines and wide size distribution and fouling of the vessel and heat transfer surface. So, these are some of the problems which can be solved by properly controlling the supersaturation and magma density and by adjusting the average residence time of the crystal.

So, we can consider that in a crystallizer if yield is less a part of mother liquor is recycled after separation of the crystals. So, that yield can be improved. A high crystal growth may adversely affect the purity since some of the foreign substances may get entrapped within the crystal. So, in that way we can control the crystal growth as well as the yield and so some problems of crystallization in industry can be resolved.

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Selection of Crystallizers		
Crystalliser type	Applications	Typical uses
Tank ✓	Batch operation, small-scale production	Fatty acids, vegetable oils, sugars ✓ ✓ ✓
Scraped surface ✓	Organic compounds, where fouling is a problem, viscous materials	Chlorobenzenes, organic acids, paraffin waxes, naphthalene, urea
Circulating magma ✓	Production of large-sized crystals. High throughput	Ammonium and other inorganic salts, sodium and potassium chlorides
Circulating liquor ✓	Production of uniform crystals (smaller size than circulating magma). High throughputs.	Gypsum, inorganic salts, sodium and potassium nitrates, silver nitrates

And here we have some points about selection of the crystallizer as we have tank scraped surface crystallizer, circulating magma means forced circulating and DBT type. And circulating liquor that is the circulating liquor crystallizer we have discussed separately. So, you can have different application where these can be applied and the product which are formed in these crystallizer.

Such as in tank we have fatty acid, vegetable oil and sugars crystals and similarly in scraped surface because it is used for viscous material and fouling fluid it is used to make chlorobenzene organic acids, paraffin waxes, naphthalene, urea which are more prone for fouling. And similarly we have circulating magma which is used for ammonium and other inorganic salts.


And similarly for circulating liquid we prepare gypsum, sodium and potassium nitrates, silver nitrates etcetera. So, in this way we can select the suitable crystallizer depending upon the application.

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References

1	Dutta, B.K., "Principles Of Mass Transfer And Separation Processes", 2009, PHI Learning Private Limited, New Delhi.
2	Sinnott, R.K., "Coulson and Richardson's Chemical Engineering Series: Chemical Engineering Design", Vol. VI, 4 th Ed., 2005, Elsevier Butterworth-Heinemann.
3	Serth, R.W., "Process Heat Transfer: Principles and Applications" 2007, Elsevier Ltd.
4	McCabe, W.L., Smith, J.C. and Harriott, P., "Unit Operations Of Chemical Engineering" 5 th Ed., 1993, McGraw-Hill Inc., New York.



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And here we have some of the references which you can go through to have deep knowledge about the topic.

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Summary of the video

- ✓ Design of Crystallizer is illustrated with example. ✓
- ✓ Different design parameters are computed and based on these drawing of crystallizer is shown.
- ✓ Types of industrial crystallizers are detailed. ✓

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And here I have the summary of the video and this summary includes the summary of the previous video that is 41st lecture and this lecture that is 42nd. So, the summary of these two lecture is here we have design the crystallizer and that will be illustrated with the help of the examples, different design parameters are computed and based on this drawing of crystallizer is shown.

And finally we have discussed industrial crystallizers which are used and according to the application you can also choose the right crystallizer. So, that is all for now. Thank you.